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THE APPLICATION OF SOLID PHASE EXTRACTION TO THE ANALYSIS OF CHLOROPHENOLS AND PHENOXY-ACID HERBICIDES

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The liquid/liquid extraction procedure currently used for the extraction of organics from water is expensive and labor intensive. An alternative to liquid/liquid extraction is a technique referred to as SPE: solid phase extraction. This procedure involves a water sample passing directly through a modified silica column. The column is dried and eluted with an organic solvent. Thus, the extraction, concentration, and cleanup are accomplished in one step. If this technique could be applied to environmental samples it would greatly improve the cost effectiveness of the analysis. A joint Ministry of the Environment/Paracel Laboratories research project was initiated to investigate thed application of solid phase extraction to the extraction of chlorophenols and phenoxy acids from water.

The study consisted of six parts:

1. A thorough literature search of solid phase extraction applications to chlorophenols and phenoxy acid analysis of water was conducted, evaluated and reported. The sources searched were Chemical Abstracts, current journals and manufacturer's application notes. The information gleaned from the literature search was used to plan the subsequent stages of the study.

2. The optimum solid phase and eluting solvent combination was determined. The differences between the phases

was not as pronounced as had been anticipated, the C-18 and phenyl modified silica adsorbents were approximately equivalent with respect to the efficiency of recovery of the chlorophenols and phenoxy acids studied, and the cyclohexyl and cyanopropyl were marginally poorer.

Iso-octane was not sufficient to elute the majority of components satisfactorily. Ethyl acetate was superior and was used for the balance of the study.

3) The breakthrough volumes of the various chlorophenols and phenoxyacids were investigated on the different phases. It was found that 100 ml of surface water could be passed through the 100 mg column and still recover most analytes satisfactorily volumes of 300 ml did not appear to wash additional material off the column.

4) Solid phases manufactured by different companies were compared and the most efficient were selected. Significant variation between manufacturers exists with respect to efficiency of recovery of chlorophenols and phenoxy acids for similar adsorbents.

5) The recoveries at different concentrations and in different aqueous matrices (low and high humic acid content, river and tap waters) were determined. Recoveries were generelly reproducible over a range of concentrations and a variety of surface and potable waters.

6) The volume of different water samples, that could

be passed through the column before plugging occurred, was determined. Volumes greater than 100 ml of water caused significant slowing of the flow rates, volumes above 200-300 mls appear impractical for the 100 mg tube.

With further study and improvement this should prove to be a rapid reliable method for extracting and concentrating these compounds. Attempts are currently being made to implement this method in MOE laboratories.